

ANALYST:	FACILITY No.
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**Parameter: Anions by Ion Chromatography (IC)**

**Method: EPA Method 300.0 Rev. 2.1**

5/04

## EQUIPMENT

- 1) Is the analytical balance capable of accurately weighing to the nearest 0.0001g? (6.1)
- 2) Does the Ion chromatograph system have an anion analytical column that gives comparable peak resolution to that of a Dionex AS4A column (P/N 37041)? (6.2.2.1)
- 3) Is IC system equipped with an anion suppressor device? (6.2.3)
- 4) Is the IC system detector a conductivity cell with an approximate internal volume of 1.25 µL? (6.2.4)
- 5) If a stripchart recorder and integrator or computer based data system other than Dionex AI-450 Data Chromatography Software is used to generate data, were MDL's approximately the same as given in the method? Nitrite - 0.004 mg/L; Nitrate - 0.002 mg/L; O-Phos - 0.003 mg/L; Sulfate - 0.02 mg/L (6.3)

## REAGENTS AND STANDARDS

- 6) Is eluent solution prepared by dissolving 0.2856 g sodium bicarbonate (CAS 144-55-8) and 0.3816 g of sodium carbonate (CAS 497-19-8) in reagent water and diluting to 2 L? (7.3)
- 7) Is the regeneration solution (micro membrane suppressor) prepared by diluting 2.8 mL conc. sulfuric acid to 4 L with reagent water? (7.4)
- 8) Are stock standards within expiration dates? Manually prepared stocks are stable for at least one month when stored at 4°C. (7.5)
- 9) Are working standards containing nitrite or phosphate prepared fresh daily and others prepared weekly? (7.5)

## SAMPLE PREP

- 10) Are samples, other than those being analyzed only for chloride, preserved by cooling to 4°C? (40 CFR, Part 136)
- 11) Is holding time for nitrate, nitrite and O-Phosphate 48 hours? (40 CFR, Part 136)
- 12) For chloride, combined nitrate/nitrite and sulfate, is holding time 28 days? (40 CFR, Part 136)
- 13) If the determined value for the combined nitrate/nitrite exceeds 0.5 mg/L as N, is sample reanalyzed for the individual concentrations of nitrate and nitrite? (8.2)

## QUALITY CONTROL

- 14) Have the following Initial Demonstration of Performance (IDP) items been completed? (9.1)
  - a. Linear Calibration Range (LCR) must be determined initially and every 6 months. LCR consists of at least one blank and three standards. If any portion is nonlinear, additional standards must be used to clearly define the nonlinear portion (9.2.2)
  - b. Quality Control Sample (QCS) must be analyzed initially and at least quarterly with 90-110% recovery. (9.2.3)
  - c. Method Detection Limit (MDL) must be determined initially, every six months, when a new operator begins work, or if there has been a significant change in the background or instrument response. (9.2.4)
- 15) Is a laboratory reagent blank (LRB) with a value below the MDL analyzed with each batch of samples? (9.3.1)
- 16) Is a laboratory fortified blank (LFB) with 90-110% recovery analyzed with each batch of samples? (9.3.2)

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17) Is a Instrument Performance Check Solution (mid-range standard with 90-110% recovery) and calibration blank analyzed immediately following the daily calibration, after every tenth sample and at the end of the sample run? (9.3.4)		
18) Is a laboratory fortified matrix (LFM) performed on 10% of routine samples with the same spike amount as the LFB? (Recovery of each analyte of interest must be 80-120% UNTIL control charts are established. Once established, control chart limits must be followed.) (9.4.1 & 9.4.3)		
19) If the identification of a peak is in doubt, are confirmatory techniques (sample dilution and fortification) used? (9.4.7)		
20) Are replicate LFBs analyzed quarterly? (9.4.8)		
<b><u>CALIBRATION</u></b>		
21) Are responses (peak height or area) tabulated against concentration to establish the calibration curve? (10.3)		
22) Are retention times recorded for calibration and samples? (10.3)		
23) Is calibration curve verified on each working day, or whenever anion eluent is changed, and after every 20 samples? Response or retention time for any analyte must be 90-110% of the expected value.(10.4)		
24) If the calibration verification is greater than $\pm 10\%$ , are fresh calibration standards used to repeat the test? (10.4)		
25) If second calibration verification fails, is a new calibration curve prepared? (10.4)		
26) Is same size loop and fixed volume used for samples and standards? (11.3)		
<b><u>PROCEDURE</u></b>		
27) Is sample mixed prior to injection? (11.3)		
28) Are sample values bracketed by calibration standards? (11.5)		
29) If interference problems (overlapping peaks) occur, is the sample diluted and/or spiked? (4.1)		

<b>PROBLEMS:</b>	NONE
<b>Comments:</b>	